

P21479.A01

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: N. ERGUN et al.

Group Art Unit: UNKNOWN

Serial No.: Not yet assigned

Examiner: UNKNOWN

Filed: Concurrently herewith

For: METHOD FOR PRODUCING FATTY ACID METHYL ESTER AND
EQUIPMENT FOR REALISING THE SAME

PRELIMINARY AMENDMENT

Assistant Commissioner of Patents
Washington, D.C. 20231

Sir:

Prior to an examination of the above-identified patent application, the Examiner is respectfully requested to amend the application as follows:

IN THE SPECIFICATION

Please amend the specification as follows (a marked-up copy of changes to the specification is attached to the present amendment):

Page 2, replace the first paragraph with the following new paragraph:

---From Falbe and Regitz, RÖMPP Chemie Lexikon, 9th Edition, Vol. 2, Georg Thieme Verlag Stuttgart-New York 1990, page 1343, a method for the production of fatty acid methyl ester is known, wherein distillation is carried out after sedimentation of the glycerine solution in a separator, in order to purify and fractionate the methyl ester if required. Moreover, the reaction rate of the transesterification can be accelerated by increasing the temperature and with the help of alkaline or acid catalysts. The disadvantage of this method is the fact that the precipitation phase of

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the glycerine solution in the separator and even the possibility to accelerate the reaction time does not shorten the production time notably compared with the above state of the art.---

Page 2, replace the second paragraph with the following new paragraph:

---Moreover, AT-PS 398 777 contains a method for the cleaning of raw vegetable oil esters, whereby the vegetable oil ester is obtained by alkaline transesterification. The transesterification takes place with methanol in excess, with the addition of potassium hydroxide as a catalyst. The raw vegetable oil ester is treated with water vapor, whereby a glycerine phase is produced, which is removed. In this process, intensive mixing is required for transesterification, and the distilled alcohol can be recycled after recovery in a distillation column.---

Page 2, line 26, insert and center the following:

--- SUMMARY OF THE INVENTION----

Page 3, replace the first full paragraph with the following new paragraph:

---The method according to the invention is characterized by mixture boundary surfaces which are enlarged by high or dynamic turbulence.---

Page 3, replace the fourth full paragraph with the following new paragraph:

---This invention makes it possible to accelerate the reaction by enlarging the boundary surfaces with dynamic processes during the transesterification. Due to high or powerful dynamic turbulence, the size of the drops in the liquid phases is effectively reduced, so that much smaller drops are produced, resulting in a much larger surface, which means that the chemical balance state is reached faster. Reaching the chemical balance state may take less than a minute. This means an enormous shortening of the production time. However, the method according to this invention is not

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suited for the so-called sedimentation method, since the sedimentation times would be too long due to the fine distribution of the drops.---

Replace the paragraph bridging pages 3 and 4 with the following new paragraph:

---According to a special feature of the invention, the high or powerful dynamic turbulence is produced by physical forces, for example mechanical shear forces. The advantage thereby is that the shear forces, which can certainly be created mechanically, produce much more powerful turbulence, thus increasing the number of drops and reducing the drop size.---

Page 4, replace the third full paragraph with the following new paragraph:

---In accordance with a further feature of the invention, the high or powerful turbulence is created by cavitation emulsification. With this process step, the reaction time can be shortened even further, since it takes only about 20 seconds with this optimization.---

Replace the paragraph bridging pages 4 and 5 with the following new paragraph:

---In accordance with another feature of the invention, transesterification starts under high pressure, and the pressure is reduced during the transesterification. It is proven that the shorter the length of the reaction section, the greater the pressure loss. It is proven that the pressure loss benefits the enlargement of the boundary surfaces. The advantage thereby is that the pressure at the beginning of transesterification can be up to 200 bar. Naturally, this increases the dynamics of the turbulence. The pressure loss is transformed into an enlargement of the boundary surfaces and dynamics in the course of the reaction section.---

Page 7, replace the second full paragraph with the following new paragraph:

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---The aim of the invention is realized independently by an equipment for implementation of the process. The equipment according to the invention is characterized by the fact that it includes at least one container for the fats, and at least one tank each for the potent alkaline solution and the alcohol, as well as at least one mixing vessel for compounding, and that at least the container and the mixing vessel are connected to the reaction section, and that there is a unit for separating the phases of the emulsion downstream from the reaction section. With this equipment according to the invention, it is for the first time possible to realize the process according to the invention in an efficient manner and with negligible environmental pollution. The equipment according to the invention offers the advantage that it can be built in a space-saving and thus cost-efficient manner. Construction as a major industrial plant for economic operation is possible.---

Page 7, replace the third full paragraph with the following new paragraph:

---In accordance with a special feature of the invention, the reaction section comprises a static mixer. With this design of the invention, high or powerful dynamic turbulence for the transesterification phase can be achieved with a simple device. This standard device has proven advantageous in use with the equipment according to the invention.---

Page 7, replace the fourth full paragraph with the following new paragraph:

---In accordance with another design of the invention, the static mixer preferably comprises a pipe filled with balls of various size and/or possibly with devices such as baffles, propellers, resistors, etc. This device is easy to install and does not require much maintenance during operation. The turbulence is created primarily by the rapid flow of the mixture around the balls.---

Page 8, replace the first paragraph with the following new paragraph:

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---In accordance with another feature of the invention, a dynamic emulsifier is included in the reaction section. Since an emulsion is to be produced in the reaction section, i.e., a liquid with two phases maintained in suspension by Braun molecular movement, such a device is particularly suitable for reaching this objective very quickly.---

Page 8, replace the fourth paragraph with the following new paragraph:

---In accordance with a special design of the invention, a mixed form of crack emulsifier and turbulator is included in the reaction section, for example, comprising two discs moving in relation to one another, whereby the emulsion is introduced in the middle of one of the discs. The advantage of such a device can be seen in the extremely short reaction time thus possible.- --

Page 9, replace the second full paragraph with the following new paragraph:

---In accordance with a further feature of the invention, the surface filter comprises a porous carrier and a layer applied to this carrier that acts as a membrane. With such an advantageous surface filter, the plant can be operated with maximum efficiency and making best use of energy. The surface filter can, of course, also be designed as a plate.---

Page 9, replace the fifth full paragraph with the following new paragraph:

---In accordance with a further feature of the invention, the layer acting as a membrane has lipophilic and/or hydrophilic and/or amphoteric properties. With the choice of these properties, it is possible to determine which phase of the mixture passes through the filter and which phase remains as residue.---

Page 9, replace the sixth full paragraph with the following new paragraph:

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---In accordance with a special feature of the invention, the layer acting as a membrane is a ceramic membrane made, for example, of titanium dioxide, zirconium dioxide, silicon or silicon compounds, etc. On this layer with the cited materials, a covering layer of fatty acid methyl ester forms, which will not allow the glycerine phase to pass through, for example. A glycerine layer will not form as a covering layer on this layer of titanium dioxide or zirconium dioxide.---

Page 10, replace the first paragraph with the following new paragraph:

---In accordance with one design of the invention, the layer acting as a membrane has a pore size in the nano and/or micro range, in particular with a size of 1-200 nm. Such pore sizes can be produced with modern technology, and have delivered excellent results.---

Page 10, replace the fourth paragraph with the following new paragraph:

---In accordance with a special feature of the invention, a distillation unit comprising at least one evaporator and one condenser is included downstream from the reaction section, or possibly downstream from the phase separation unit. This unit according to the invention also makes it possible to realize the process according to the invention in an efficient manner.---

Page 10, replace the fifth paragraph with the following new paragraph:

---In accordance with a further feature of the invention, a down-flow evaporator is used as an evaporator. Such an evaporator has the advantage that the supplied heat can be used optimally. Evaporation in a vacuum is also possible.---

Page 10, replace the sixth paragraph with the following new paragraph:

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---In accordance with another design of the invention, a thin-layer evaporator is used as an evaporator. Evaporation in a vacuum is also possible with such an evaporator. In addition, optimal results are obtained with such a device.---

Page 10, replace the seventh paragraph with the following new paragraph:

---In accordance with a special design of the invention, a rotary flow evaporator is used as an evaporator. Due to the centrifugal force of the rotary flow evaporator, the evaporator film is particularly thin-layered, so that the plant can be operated with maximum efficiency and making optimal use of energy.---

Page 11, line 21, insert and center the following:

---BRIEF DESCRIPTION OF THE DRAWINGS---

Page 11, replace the seventh paragraph with the following new paragraph:

---Fig. 1 is a flow chart of the process flow with a distillation unit, and---

Page 11, replace the eighth paragraph with the following new paragraph:

---Fig. 2 is a flow chart of the process flow with a filtration unit.---

Page 12, before line 1, insert and center the following:

--- DETAILED DESCRIPTION----

Page 13, replace the fourth paragraph with the following new paragraph:

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---The distillation unit 15 comprises at least one evaporator and one condenser, whereby the distillation unit 15 is designed as a vacuum distillation unit. The target substances, such as the fatty acid methyl ester, are correspondingly removed from the stages 18 of the distillation unit 15.---

Page 13, replace the fifth paragraph with the following new paragraph:

---Of course, various forms of an evaporator can be used. For example, a down-flow evaporator, thin-layer evaporator, rotary flow evaporator, etc. can be used. In addition, the distillation unit 15 also comprises fractionated condensation. With the design of the distillation unit 15, the degree of purity of the fatty acid methyl ester can be influenced.---

Page 13, replace the sixth paragraph with the following new paragraph:

---Non-transesterified substances are separated and collected in a separating unit 19, which is connected to the distillation unit 15 by a pipeline 20. A pump 21 is provided in the pipeline 20 for introducing these substances to the separating unit 19. These parts are analyzed in the separating unit 19 and then accordingly processed further. If necessary, part of the non-transesterified substances are returned to the fats upstream from the reaction section 8 via a pipeline 22 with a pump 23. Certain substances are removed from the separating unit 19 by a recovery unit.---

Page 15, replace the second paragraph with the following new paragraph:

---The unit 27 comprises at least one filtration unit 29. In accordance with the design of the unit 27, the individual phases, e.g., the fatty acid methyl ester, are separated and removed at the recovery point 30. The glycerine phase can be removed for further use at the recovery point 31.---

Page 15, replace the fourth paragraph with the following new paragraph:

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---The filtration unit 29 is a surface filter manufactured using membrane technology and comprising a porous carrier, for example of aluminum oxide, preferably designed as a pipe, and a layer applied to the carrier, for example a layer of titanium dioxide. The layer can have amphoteric properties, whereby the pore size is in the nano range.---

Page 15, replace the fifth paragraph with the following new paragraph:

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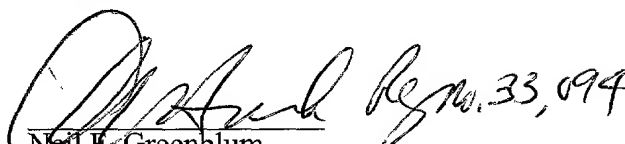
REMARKS

The Examiner is respectfully requested to enter the foregoing amendment prior to examination of the above-identified patent application. The present amendment includes changes made in the parent application.

Should there be any questions, the Examiner is invited to contact the undersigned at the below-listed telephone number.

September 27, 2001
GREENBLUM & BERNSTEIN, P.L.C.
1941 Roland Clarke Place
Reston, VA 20191
(703)716-1191

Respectfully submitted,
N. ERGUN et al.


Neil F. Greenblum
Reg. No. 28,394

MARKED-UP COPY OF CHANGES TO SPECIFICATION:

Page 2, replace the first paragraph with the following new paragraph:

---From [the literature search in] **Falbe and Regitz, RÖMPP Chemie Lexikon**, 9th Edition, Vol. 2, Georg Thieme Verlag Stuttgart-New York 1990, page 1343, a method for the production of fatty acid methyl ester is known, wherein distillation is carried out after sedimentation of the glycerine solution in a separator, in order to purify and fractionate the methyl ester if required. Moreover, the reaction rate of the transesterification can be accelerated by increasing the temperature and with the help of alkaline or acid catalysts. The disadvantage of this method is the fact that the precipitation phase of the glycerine solution in the separator and even the possibility to accelerate the reaction time does not shorten the production time notably compared with the above state of the art.---

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---Moreover, AT-PS 398 777 contains a method for the cleaning of raw vegetable oil esters, whereby the vegetable oil ester is obtained by alkaline transesterification. The transesterification takes place with methanol in excess, with the addition of potassium hydroxide as a catalyst. The raw vegetable oil ester is treated with water [vapour] vapor, whereby a glycerine phase is produced, which is removed. In this process, intensive mixing is required for transesterification, and the distilled alcohol can be recycled after recovery in a distillation column.---

Page 2, line 26, insert and center the following:

--- SUMMARY OF THE INVENTION----

Page 3, replace the first full paragraph with the following new paragraph:

---The method according to the invention is [characterised] characterized by [the fact that the] mixture boundary surfaces [of the mixture] which are enlarged by high or dynamic turbulence.---

Page 3, replace the fourth full paragraph with the following new paragraph:

---This invention makes it possible to accelerate the reaction by enlarging the boundary surfaces [, and] with dynamic processes during the transesterification. Due to [the] high or powerful dynamic turbulence, the size of the drops in the liquid phases is effectively reduced, so that much smaller drops are produced, resulting in a much larger surface, which means that the chemical balance state is reached faster. Reaching the chemical balance state may take less than a minute. This means an enormous shortening of the production time. However, the method according to this invention is not suited for the so-called sedimentation method, since the sedimentation times would be too long due to the fine distribution of the drops.---

Replace the paragraph bridging pages 3 and 4 with the following new paragraph:

---According to a special feature of the invention, the high or powerful dynamic turbulence is produced by physical forces, for example mechanical shear forces. The advantage thereby is that the shear forces, which can certainly be created mechanically, produce much more powerful turbulence, thus increasing the number of drops [at the expense of] and reducing the drop size.---

Page 4, replace the third full paragraph with the following new paragraph:

---In accordance with a further feature of the invention, the high or powerful turbulence is created by cavitation emulsification. With this process step, the reaction time can be shortened even further, since it takes only about 20 seconds with this [optimisation] optimization.---

Replace the paragraph bridging pages 4 and 5 with the following new paragraph:

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---In accordance with another feature of the invention, transesterification starts under high pressure, and the pressure is reduced during the transesterification. It is proven that the shorter the length of the reaction section [can be all the shorter], the greater the pressure loss [is]. It is proven that the pressure loss benefits the enlargement of the boundary surfaces. The advantage thereby is that the pressure at the beginning of transesterification can be up to 200 bar. Naturally, this increases the dynamics of the turbulence. The pressure loss is transformed into an enlargement of the boundary surfaces and dynamics in the course of the reaction section.---

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the transesterification phase can be achieved with a simple device. This standard device has proven advantageous in use with the equipment according to the invention.---

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---In accordance with a further feature of the invention, the surface filter [consists of] comprises a porous carrier and a layer applied to this carrier that acts as a membrane. With such an

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advantageous surface filter, the plant can be operated with maximum efficiency and making best use of energy. The surface filter can, of course, also be designed as a plate.---

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---In accordance with a special feature of the invention, a distillation unit [consisting of] comprising at least one evaporator and one condenser is included downstream from the reaction section, or possibly downstream from the phase separation unit. This unit according to the invention

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also makes it possible to [realise] realize the process according to the invention in an efficient manner.---

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Page 10, replace the sixth paragraph with the following new paragraph:

---In accordance with another design of the invention, a thin-layer evaporator is used as an evaporator. Evaporation in a vacuum is also possible with such an evaporator. In addition, optimal results are obtained with such a device.---

Page 10, replace the seventh paragraph with the following new paragraph:

---In accordance with a special design of the invention, a rotary flow evaporator is used as an evaporator. Due to the centrifugal force of the rotary flow evaporator, the evaporator film is particularly thin-layered, so that the plant can be operated with maximum efficiency and making optimal use of energy.---

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Page 12, before line 1, insert and center the following:

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Page 13, replace the fourth paragraph with the following new paragraph:

---The distillation unit 15 [consists of] comprises at least one evaporator and one condenser, whereby the distillation unit 15 is designed as a vacuum distillation unit. The target substances, such as the fatty acid methyl ester, are correspondingly removed from the stages 18 of the distillation unit 15.---

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---Of course, various forms of an evaporator can be used. For example, a down-flow evaporator, thin-layer evaporator, rotary flow evaporator, etc. can be used. In addition, the distillation unit 15 also comprises fractionated condensation. With the design of the distillation unit 15, the degree of purity of the fatty acid methyl ester can be influenced.---

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